



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of

Yuki SASAKI et al.

Appln. No.: 10/015,611

Examiner: G. Mitchell

Filed: December 17, 2001

Group Art Unit: 1617

FOR: RESIN POWDER FOR DERMATOLOGIC COMPOSITION, SKIN  
CLEANSING AGENT AND COSMETIC COMPOSITION USING THE  
POWDER, AND PREPARATION PROCESS OF THE POWDER

DECLARATION UNDER 37 C.F.R. §1.132

Honorable Commissioner for Patents  
Alexandria, VA 22313-1450

Sir/Madam:

I, Hirotaka Matsuoka, a citizen of Japan, hereby declare and state:

I have a degree in Physical Chemistry which was conferred upon me by the Faculty of Engineering of Yokohama National University in Yokohama, Japan in 1983.

I have been employed by Fuji Xerox since April of 1983 and I have had a total of 21 years of work and research experiences in research and development relating to fine particles.

I have reviewed the above-captioned U.S. Patent Application and the November 17, 2004 Office Action.

EXPERIMENTATION

1. Preparation of Colorant Dispersion

In a flask, 20 parts by weight of a zinc oxide (white colorant, FINEX 25, 0.06 µm), 2 parts by weight of an anionic surfactant (NEOGEN R, manufactured by Daiichi Pharmaceutical Co., Ltd.) and 78 parts by weight of deionized water were

mixed using a homogenizer (ULTRA TURRAX T50, product of IKA Works) at 3000 rpm for 2 minutes, then further dispersed at 5000 rpm for 10 minutes. Then the dispersion was deformed by stirring the dispersion using a commonly-used stirring apparatus for a day. The deformed dispersion was further dispersed using a high-pressure-impact dispersion apparatus (ULTIMIZER HJP30006, product of SUGINO MACHINE LIMITED) at 240MPa for about 1 hour to obtain a white colorant dispersion. The colorant dispersed in the white colorant dispersion had a number average particle size of 98 nm. Then, deionized water was added thereto to adjust a solid content concentration thereof at 20 %.

## 2. Preparation of Resin Powder

A resin powder was prepared by using the white colorant dispersion and the resin dispersion 1 used in Examples of the present application. The details are set forth below.

In an round-bottom stainless flask, 520 g of the resin dispersion 1 of the present application, 6.0 g of a 10 wt.% aqueous solution of poly(aluminum chloride) (PAC100W, product of Asada Chemical), 38 g of 0.02M nitric acid and 200 g of the white colorant dispersion were mixed and dispersed using a homogenizer (ULTRA TURRAX T50, product of IKA Works). The dispersion was heated to 60°C, with stirring, over an oil bath. After 30 minutes, 200 g of the resin dispersion was gradually added. The temperature of the oil bath was raised to 90°C, for a predetermined time (spherization time), and agglomerated particles were obtained.

52 g of 1N sodium hydroxide was then added to the flask, which was than hermetically sealed using a magnetic seal. The mixture was heated to 96°C with continued stirring. The agglomerated particles were fused by maintaining the mixture at 96°C for 7 hours. Then fused particles were obtained. The fused particles were washed with deionized water (pure water) of pH 6.5. After vacuum lyophilization, the particles were

shifted using a 20- $\mu\text{m}$  mesh to yield a resin powder.

### 3. Evaluation

The resin powder thus obtained was subjected to the measurement in the same manner as described in the present specification.

The measured value of average volume particle size, average volume particle size distribution GSDv, shape factor SF1, surfaceness index, average number particle size distribution GSDp, a ratio of particles having a volume particle size of 20  $\mu\text{m}$  or greater, number-average molecular weight, weight-average molecular weight, glass transition temperature, compaction ratio, water content, volatile content, acid value, surface tension and conductivity are shown in the following Table A.

Table A

	Resin powder of this Experimentation
Resin dispersion	1
Sphertizing time	7
Washing with pure water	done
Shifting through 20 µm sieve	done
Vacuum drying time (h)	8
Average volume particle size (µm)	9.8
Average volume particle size distribution GSDv	1.22
Shape factor SP1	112
Surfacteness Index	1.41
Average number particle size distribution GEDp	1.26
Ratio of particles having a volume particle size of 20 µm or greater (%)	0.05
Number-average molecular weight	$1.1 \times 10^4$
Weight-average molecular weight	$2.8 \times 10^4$
Glass transition temperature (°C)	71
Compaction ratio	0.38
Water content (wt. %)	0.25
Volatile content (ppm)	29
Acid value (mg/KOH/g)	5
Surface tension (mN)	45
Conductivity ( $\mu\text{S}$ )	52
Polymer	St / Ac
Preparation method	Emulsion Polymerization and Agglomeration method.
Smoothness	2.1
Spreadability	3.4
Affinity to the skin	3.3
Odor	4.1

Furthermore, the resin powder thus obtained, talc, mica, titanium oxide, mica titanium, yellow oxide, black oxide, squalane, vaseline, perfume and antiseptic were added in amounts as shown in Table B set forth below, respectively. Components Nos. 1 to 7 in Table B were mixed in a Henschel mixer. After addition of a mixture of Components Nos. 8 to 11 (refer to Table B), they were mixed uniformly, followed by pulverization. The particles thus obtained were formed into a solid powder foundation.

The solid powder foundation thus obtained was subjected to the measurement in the same manner as described in the present specification. The measured value of smoothness, spreadability and affinity to the skin are also shown in the following Table B.

Table B

1	Talc	14
2	Mica	30
3	Titanium oxide	15
4	Mica titanium	3
5	Yellow oxide	1.8
6	Black oxide	0.2
7	Resin Powder of this Experimentation	30
8	Squalane	5
9	Vaseline	2.0
10	Perfume	0.1
11	Antiseptic	0.2
	Smoothness	2.4
	Spreadability	4.0
	Affinity to the skin	3.1

As mentioned above, affinity of the resin particle having a coloring agent was impaired due to the precipitated coloring agent having a higher hardness. Furthermore, spreadability and smoothness thereof also was impaired due to the hardness and the shape of the desorbed coloring agent.

I declare further that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Date : 05/31/12

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Hirotaka MATSUOKA